



Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 797 (1982): Common salt for chemical industries [CHD 1:
Inorganic Chemicals]

“ज्ञान से एक नये भारत का निर्माण”

Satyanaaran Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”



BLANK PAGE



PROTECTED BY COPYRIGHT

IS : 797 - 1982
(Reaffirmed 2012)

Indian Standard
**SPECIFICATION FOR COMMON SALT
FOR CHEMICAL INDUSTRIES**
(Third Revision)

Fourth Reprint DECEMBER 2006
(Including Amendment No. 1)

UDC 54 - 38 : 66

© Copyright 1983

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7

April 1983

AMENDMENT NO. 1 NOVEMBER 985

TO

IS:797-1982 COMMON SALT FOR CHEMICAL INDUSTRIES

(*Third Revision*)

(Page 5, clause 4.1) - Substitute the following for the existing clause:

'4.1 Packing - The grade 1 material shall be supplied in polyethylene lined jute bags or with a liner; polyethylene lined HDPE bag or with a liner or any other suitable packing as agreed to between the purchaser and the supplier. Grade 2 material shall be packed in jute bags or any other packing as agreed to between the purchaser and the supplier or loose in truck loads country craft or steamers. The packages shall be securely closed.'

(CDC 56)

Indian Standard

**SPECIFICATION FOR COMMON SALT
FOR CHEMICAL INDUSTRIES**

(Third Revision)

Acids, Alkalies and Halides Sectional Committee, CDC 56

Chairman

PROF M. M. TAQUI KHAN

Representing

Central Salt & Marine Chemicals Research Institute
(CSIR), Bhavnagar

Members

DR G. D. BHAT (*Alternate to*
Prof M. M. Taqui Khan)

Directorate General of Supplies & Disposals, New
Delhi

SHRI S. K. BASU

National Test House, Calcutta
Directorate General of Technical Development
New Delhi

SHRI D. S. CHOWDHURY (*Alternate*)

Bengal Chemicals & Pharmaceuticals Ltd, Calcutta

SHRI M. L. BHAMBANI
SHRI R. C. BHATTACHARYA

The Atul Products Ltd, Atul

SHRI H. KHAPARDE (*Alternate*)

The Travancore Rayons Ltd, Rayonpuram
E.I.D.-Parry (India) Ltd, Madras

SHRI M. V. DESAI

DR J. M. TUREL (*Alternate*)

DR R. N. GHOSH

Ministry of Defence (DGI)

SHRI R. K. CHATTERJEE (*Alternate*)

SHRI P. S. HARIHARAKRISHNAN

Indian Paper Mills Association, Calcutta

DR H. V. R. IENGAR

DR V. C. NAIR (*Alternate*)

Indian Dyestuff Industries Ltd, Bombay

SHRI MANMOHAN SINGH

SHRI N. K. GUPTA (*Alternate*)

The Mettur Chemical & Industrial Corporation Ltd
Mettur Dam

DR B. S. NAGAR

SHRI N. R. FADNIS (*Alternate*)

The Fertilisers & Chemicals, Travancore Ltd
Udyogamandal

DR V. N. NIGAM

SHRI K. B. DUTTA (*Alternate*)

Tata Chemicals Ltd, Bombay

SHRI M. PADMANABHAN

SHRI D. PANIGRAHI

SHRI V. N. RAO

SHRI K. H. PAREKH (*Alternate*)

(Continued on page 2)

© Copyright 1983

BUREAU OF INDIAN STANDARDS

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

IS : 797 - 1982

(Continued from page 1)

Members

- SHRI B. REVENNA
SHRI N. K. SACHAR
SHRI J. B. SHARMA (*Alternate*)
SHRI C. R. SANTHANAKRISHNAN

SHRI K. V. MANI (*Alternate*)
SHRI V. S. SAXENA
SHRI R. C. SINHA (*Alternate*)
DR N. SEN

SHRI S. K. SUBBAROYAN
DR A. N. BHAT (*Alternate*)
SHRI B. K. VAHI
SHRI S. S. BANSAL (*Alternate*)
DR M. S. VAIDYA
DR A. S. VAIDYANATHAN
SHRI T. P. VENKATRAMAN (*Alt*)
SHRI H. K. VENKATARAMAIAH
DR K. M. VERMA
SHRI B. R. WALAVAIKAR
SHRI L. K. MALAVIYA (*Alternate*)
SHRI S. K. MATHUR,
Head (Chem.)

Representing

- Sa???Commissioner, Jaipur
Gujarat Alkalies & Chemicals Ltd, Vadodara

Travancore Chemical & Manufacturing Co Ltd,
Mettur Dam

Ballarpur Industries Ltd, New Delhi

Indian Soap & Toiletries Makers' Association,
Bombay

Hindustan Lever Ltd, Bombay

D. G. M. Chemical Works, New Delhi

The Dharamsi Morarji Chemical Co Ltd, Bombay
Standard Alkali, Chemicals Division, Bombay
(note)

Hindustan Organic Chemicals Ltd, Rasayani
Projects & Development India Ltd, Sindri
Saurashtra Chemicals, Porbandar
)
Director General, ISI (*Ex-officio Member*)

Secretary

- SHRI M. BAKSHI GUPTA
Assistant Director (Chem.), ISI

Common Salts & Marine Chemicals Subcommittee, CDC 56 : 4

Convener

- DR N. N. SHARMA Tata Chemicals Ltd, Bombay

Members

- SHRI K. H. PAREKH (*Alternate* to
Dr N. N. Sharma)
SHRI T. N. BALAKRISHNAN The Mettur Chemical & Industrial Corporation Ltd,
Mettur Dam
SHRI D. S. CHADHA Central Committee for Food Standards, Directorate
General of Health Services, New Delhi
SMT DESI MUKHERJEE (*Alternate*)
SHRI M. V. DESAI The Atul Products Ltd, Atul
DR J. M. TUREL (*Alternate*)
DR M. J. MEHTA Central Salt & Marine Chemicals Research Institute
(CSIR), Bhavnagar
DR J. R. SANGHAVI (*Alternate*)
DR S. K. SAGAR Shriram Chemical Industries, Kota
SHRI RAJIV SINHA (*Alternate*)

(Continued on page 28)

Indian Standard

SPECIFICATION FOR COMMON SALT FOR CHEMICAL INDUSTRIES

(*Third Revision*)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Indian Standards Institution on 31 August 1982, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1955 and subsequently revised in 1967. The original version included only one grade of the material. In the first revision another grade of the material required for the manufacture of hydrosulphite and optical brighteners was incorporated. The standard was again revised in 1976; in the second revision the requirements of magnesium and sulphate contents had been modified. In grade 2 of the material, optional requirements for grit content and trace metals like vanadium, chromium, titanium, molybdenum, copper, nickel and aluminium were incorporated for use of the material in the explosive and pyrotechnic industry and electrolytic purposes respectively. The methods of test for the determination of the trace metals were also incorporated. The sampling scheme was also modified accordingly.

0.3 In the present revision, additional requirements for the materials of Grade 2, for use in the explosives and pyrotechnic industry for sulphate, iron and alkalinity have been incorporated. The method of test for the determination of alkalinity has also been incorporated.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and, test for common salt for chemical industries.

*Rules for rounding off numerical values (*revised*)

2. GRADES

2.1 There shall be two grades of the material, namely:

Grade 1 — suitable for the manufacture of hydrosulphite and optical brighteners or where a pure grade of the material is required.

Grade 2 — suitable for chemical industries, explosive and pyrotechnic industry and for the manufacture of caustic soda by electrolytic process.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of large or small colourless crystals, or a white granular powder. The material shall be free from visible contamination with clay, grit and other extraneous adulterants and impurities.

3.2 Moisture Content

3.2.1 For Grade 1 Material — It shall contain not more than 1.0 percent moisture, on as received basis, when tested in accordance with the method prescribed in **A-2**.

3.2.2 For Grade 2 Material — It shall contain not more than 4.0 percent moisture, on as received basis, when tested in accordance with the method prescribed in **A-2**.

3.3 The material shall also comply with the requirements specified in Table 1 when tested according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR COMMON SALT FOR CHEMICAL INDUSTRIES

SL NO.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Grade 1 (On Dry Basis)	Grade 2 (On Dry Basis)	
(1)	(2)	(3)	(4)	(5)
i)	Sodium chloride (as NaCl), percent by mass, <i>Min</i>	99.5	98.5	A-3
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.05	0.2	A-4
iii)	Calcium salts (as Ca), percent by mass, <i>Max</i>	0.03	0.2	A-5
iv)	Magnesium salts (as Mg), percent by mass, <i>Max</i>	0.01	0.10	A-5
v)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.2	0.6	A-6
vi)	Iron compound (as Fe), parts per million, <i>Max</i>	10	20	A-7

3.3.1 Optional Requirements

3.3.1.1 For use in the explosives and pyrotechnic industry, the material of Grade 2 shall have grit content of 20 ppm maximum when tested in accordance with the method prescribed in **A-11**.

For use in the explosives and pyrotechnic industry, the material of Grade 2 shall also have the following additional requirements when tested in accordance with the methods prescribed in **A-6**, **A-7** and **A-12**.

- a) Sulphate — 0.5 percent, *Max*,
- b) Iron — 10 parts per million, and
- c) Alkalinity — 0.5 percent, *Max*
(as Na_2CO_3)

3.3.1.2 For the manufacture of caustic soda by the electrolytic process, the material of Grade 2 shall have the following additional requirements when tested in accordance with the methods prescribed in **A-8**, **A-9** and **A-10** of Appendix A:

	<i>Parts per Million, Max</i>
a) Vanadium + chromium + molybdenum	0.01
b) Aluminium (as Al)	0.10
c) Other trace metals like titanium, copper and nickel	0.01

4. PACKING AND MARKING

4.1 Packing— The Grade 1 material shall be supplied in polyethylene lined jute bags and Grade 2 shall be packed in jute bags or loose in truck loads, country craft or steamers. The packages shall be securely closed.

4.2 Marking— The packages shall be marked legibly and indelibly with the following information:

- a) Name and grade of material;
- b) Mass of the material;
- c) Lot or batch number in code or otherwise; and
- d) Name of manufacturer and recognized trade-mark; if any.

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of preparation of test samples and determination of their criteria for conformity shall be as given in Appendix B.

APPENDIX A

(*Clauses 3.2.1, 3.2.2, 3.3, 3.3.1.1, 3.3.1.2 and Table 1*)

METHOD OF TEST FOR COMMON SALT FOR CHEMICAL INDUSTRIES

A-0. GENERAL

A-0.1 Quality of Reagents — Unless specified otherwise, pure chemical and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-1. PREPARATION OF SAMPLE

A-1.1 Crush 500 g of the sample to pass through a 1-mm IS Sieve, dry in an air-oven at about 150°C for at least 4 hours. Cool in a desiccator and transfer to a clean glass-stoppered weighing bottle kept in a desiccator for subsequent tests.

A-1.2 Accurately weigh about 20 g of the dried sample, dissolve it in 200 ml of water in a beaker. Heat to boiling, filter and wash the paper free of chloride. Cool and make up the solution to 1 litre. Use this solution for subsequent tests.

*Specification for water for general laboratory use (second revision).

A-1.3 For trace metal analyses, dissolve completely 3 200 g of the salt samples in water, acidify with 100 ml hydrochloric acid and stir well to dissolve all the soluble matter. Filter and make up to 10 litres. Use this solution for all trace metal analyses.

A-2. DETERMINATION OF MOISTURE CONTENT

A-2.1 Procedure — Grind rapidly the material, as received (say 100 g), in an agate mortar approximately to a size of 2.8 mm IS Sieve, but do not actually sieve. The material which shall be in the form of powder shall be kept in an air-tight container. Weigh accurately about 20 g of the material in the weighing bottle of about 30 ml capacity, preferably wide mouth squat type, previously dried and weighed. Dry in an oven at 140 to 150°C for at least 4 hours. Cool in a desiccator and weigh. Repeat drying, cooling and weighing until constant mass is obtained.

A-2.2 Calculation

$$\text{Moisture, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the material after drying.

A-3. DETERMINATION OF TOTAL CHLORIDES

A-3.1 Reagents

A-3.1.1 Potassium Chromate Indicator Solution — 5 percent.

A-3.1.2 Standard Silver Nitrate Solution — 0.1 N.

A-3.2 Procedure — Transfer 10 ml of the solution reserved in A-1.2 into a conical flask and add 1 ml of potassium chromate indicator solution. Titrate against standard silver nitrate solution till the reddish brown tinge persists after brisk shaking. Carry out a blank determination.

A-3.3 Calculation

$$\text{Total chlorides (as Cl), percent by mass} = \frac{3.546 \times VN}{M}$$

where

V = volume in ml of standard silver nitrate solution used in the titration with the material corrected to blank,

N = normality of standard silver nitrate solution, and

M = mass in g of the dried sample in 10 ml of the solution taken for the test.

A-3.3.1 Residual chloride is the chloride obtained by deducting the chloride attributed to the other chlorides, such as magnesium chloride and calcium chloride as described in **A-6** of IS : 253-1970* from the total chlorides determined in **A-3.3**.

Sodium chloride (as NaCl), = Residual chloride (as Cl) \times 1.648 percent by mass

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Procedure — Accurately weigh about 20 g of the dried sample (*see A-1.1*), dissolve it in 200 ml of water in a beaker, heat to boiling and cool. Filter the solution through a weighed Gooch or sintered glass crucible (G No. 4) and wash the residue till it is free from chlorides. Dry the crucible along with the insoluble residue to constant mass.

A-4.2 Calculation

$$\text{Matter insoluble in water} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the dried sample taken for the test.

A-5. DETERMINATION OF CALCIUM AND MAGNESIUM

A-5.1 Reagents

A-5.1.1 Standard Calcium Solution — Weigh 1.000 g of calcium carbonate, dried at 120°C, and dissolve it in the minimum quantity of dilute hydrochloric acid. Dilute the solution to one litre in a graduated flask. One millilitre of the solution is equivalent to 0.400 3 mg of calcium (as Ca).

A-5.1.2 Standard EDTA Solution — Dissolve 3.72 g of disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against standard calcium solution following the procedure given in **A-5.2**.

A-5.1.3 Eriochrome Black T Indicator Solution — Dissolve 0.1 g of the dye in 20 ml of rectified spirit conforming to IS : 323-1959†. This solution shall be prepared fresh every week.

A-5.1.4 Dilute Sodium Hydroxide Solution — approximately 10 percent.

*Specification for edible common salt (*second revision*).

†Specification for rectified spirit (*revised*).

A-5.1.5 Murexide Indicator Solution — Grind 0.2 g of murexide with 10 g of sodium chloride until the mixture is homogenous. 0.2 g of this mixture is suitable for 100 ml of the sample solution.

A-5.1.6 Calcein Indicator — Grind thoroughly 0.1 g of calcein and 0.06 g of thymolphthalein with 10 g of potassium chloride.

A-5.1.7 Ammonium Chloride-Ammonium Hydroxide Buffer Solution — Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp gr 0.90) and 250 ml of water. Also dissolve separately a mixture of 0.931 g of disodium ethylene diamine tetra acetate dihydrate and 0.616 g of magnesium sulphate ($MgSO_4 \cdot 7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to one litre.

NOTE — Five millilitres of buffer solution added to 50 ml of distilled water should not consume more than a drop of EDTA solution to change to distinct blue with eriochrome black T indicator.

A-5.2 Procedure

A-5.2.1 Standardization of EDTA Solution — Transfer 25 ml of standard calcium solution into a conical flask, add 25 ml of water, 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of the eriochrome black T indicator solution and titrate against the standard EDTA solution to a pure blue end point.

A-5.2.2 Titrate 25 ml of the buffer solution with EDTA solution using eriochrome black T indicator. Subtract the buffer correction for 10 ml (usually it will be 0.1 ml) from the reading obtained in **A-5.2.1** and note the final titre value. Calculate the calcium equivalent of 1 ml of EDTA solution (say A).

A-5.2.3 Transfer exactly 100 ml of the solution preserved in **A-1.2** into a 250-ml conical flask, add 10 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end point. Note the volume of EDTA used in the titration.

A-5.2.4 Transfer exactly 100 ml of the solution preserved in **A-1.2** into 250-ml conical flask add 5 mg sodium hydroxide solution and stir well. Add 0.2 g murexide (or 100 ml calcein indicator) and titrate against standard EDTA solution till wine red colour of the solution changes to pure blue end point with murexide (or green to purple with mixed calcein indicator). Note the volume of the standard EDTA solution used in the titration.

A-5.3 Calculation

$$\text{Calcium (as Ca), percent by mass} = \frac{AV_2}{M}$$

$$\text{Magnesium (as Mg), percent by mass} = 0.6068 \frac{A(V_1 - V_2)}{M}$$

where

A = calcium equivalent in mg of 1 ml of EDTA solution determined in A-5.2.2,

V_2 = volume in ml of standard EDTA solution used in A-5.2.4 (for Ca only),

M = mass in g of the dried sample taken for the test in A-1.2, and

V_1 = volume in ml of standard EDTA solution used in A-5.2.3.

A-6. DETERMINATION OF SULPHATE**A-6.1 Gravimetric Method****A-6.1.1 Reagents**

A-6.1.1.1 Dilute hydrochloric acid — approximately 4 N.

A-6.1.1.2 Barium chloride solution — approximately 10 percent,

A-6.1.2 Procedure — Dissolve about 10 g of dried sample preserved in A-1.1 in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Add one drop of methyl orange and 10 ml of dilute hydrochloric acid or more till it is pink and then boil. Add to the boiling solution, 10 to 12 ml of barium chloride solution drop by drop so that the addition is in slight excess and continue boiling for 4 minutes to obtain a granular precipitate. Allow to stand for 4 hours and filter through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash the precipitate till free from chloride and dry to constant mass at 105 to 110°C. Alternatively, filter through Whatman filter paper No. 42 and wash till it is free from chloride. Ignite and determine as barium sulphate.

A-6.1.3 Calculation

$$\text{Sulphate (as SO}_4\text{), percent by mass} = 41.13 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of barium sulphate, and

M_2 = mass in g of dried sample taken for test.

A-6.2 Volumetric Method

A-6.2.1 Reagents

A-6.2.1.1 Standard barium chloride Solution— 0.05 N. Dissolve 6.108 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and make up to one litre.

A-6.2.1.2 Dilute hydrochloric acid— approximately 1 N.

A-6.2.1.3 Standard EDTA solution— Weigh 3.72 g of disodium ethylene diamine tetra acetate dihydrate in water and dilute in a graduated flask to one litre. The solution shall be standardized frequently against standard barium chloride solution (A-6.2.1.1) and follow the procedure given in A-5.2. One millilitre of standard EDTA solution is equal to 0.001 374 g of barium (Ba) or 0.000 96 g of sulphate (SO_4).

A-6.2.1.4 Eriochrome black T indicator solution— same as in A-5.1.3.

A-6.2.1.5 Ammonium chloride-ammonium hydroxide buffer solution— same as in A-5.1.7.

A-6.2.2 Procedure

A-6.2.2.1 Pipette out 20 ml of standard barium chloride solution into a conical flask, add 2 drops of hydrochloric acid and 10 ml of ammonium chloride-ammonium hydroxide buffer solution. Dilute it with water to about 50 ml, add five drops of eriochrome black T indicator solution and titrate against standard EDTA solution to pure blue end point. Note the titre value (say A).

A-6.2.2.2 Pipette out 100 ml of the solution preserved in A-1.2 and add two drops of hydrochloric acid and heat to gentle boiling. To the hot solution, add 20 ml of standard barium chloride solution. Boil gently for about 5 minutes and then cool to room temperature. The solution will be neutral or slightly acidic when tested with a litmus paper. Add 10 ml of ammonium chloride-ammonium hydroxide buffer solution and five drops of eriochrome black T indicator solution. Titrate against standard EDTA solution to pure blue end point. Note the titre value (say B).

A-6.2.3 Calculation

$$\text{Sulphate (as } \text{SO}_4\text{), percent} = 0.96 \frac{A + V_1 - B}{M}$$

by mass (on dry basis)

where

A = volume in ml of standard EDTA solution used in A-6.2.2.1,

V_1 = volume in ml of standard EDTA solution used in A-5.2.3
(total Ca + Mg),

B = volume in ml of standard EDTA solution used in titration
in A-6.2.2.2, and

M = mass in g of the dried sample taken for the test in A-1.2.

A-7. TEST FOR IRON

A-7.1 Apparatus

A-7.1.1 Nessler Cylinders — 50 ml capacity (see IS : 4161-1967*)

A-7.2 Reagents

A-7.2.1 Dilute Hydrochloric Acid — approximately 5 N, free from iron.

A-7.2.2 Concentrated Nitric Acid — see IS : 264-1976†.

A-7.2.3 Potassium Thiocyanate Solution — 5 percent (m/v).

A-7.2.4 Dilute Sulphuric Acid — approximately 5 N.

A-7.2.5 Standard Iron Solution — Dissolve 2.81 g of ferrous ammonium sulphate [$\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Transfer 10 ml of the solution to 1 000 ml volumetric flask and dilute again up to the mark. One millilitre of this solution is equivalent to 0.004 mg of iron (as Fe).

A-7.3 Procedure — Weigh accurately about 2 g of the material and dissolve it in about 20 ml of water. Add about 5 ml of hydrochloric acid in order to make the solution acidic and 2 ml of nitric acid, and boil to oxidize the iron salts. Cool and transfer to a Nessler cylinder, add 5 ml of potassium thiocyanate solution, dilute to the mark with water and stir well. Into a second Nessler cylinder, add 5 ml of hydrochloric acid, 2 ml of nitric acid, 5 ml standard iron solution for Grade 1 and 10 ml of standard iron solution for Grade 2 material, and 5 ml potassium thiocyanate solution. Dilute to the mark and stir well. Compare the colour of the solution in the two cylinders.

A-7.3.1 The limit prescribed in Table 1 shall be taken to have not been exceeded if the intensity of colour produced with the sample is not greater than that produced by the standard iron solution.

A-8. TEST FOR VANADIUM, CHROMIUM AND MOLYBDENUM

A-8.0 Each of these trace metals is estimated separately and the results added.

*Specification for Nessler cylinders.

†Specification for nitric acid (second revision).

A-8.1 Estimation of Vanadium

A-8.1.0 Principle — The vanadium is extracted from a large quantity of sample with 8-hydroxyquinoline, and subsequently determined colourimetrically as the phosphotungstate.

A-8.1.1 Reagents

A-8.1.1.1 8-hydroxyquinoline reagent — Dissolve 4 g of the reagent in 100 ml of 10 percent acetic acid.

A-8.1.1.2 Sodium acetate — 5 percent.

A-8.1.1.3 Chloroform

A-8.1.1.4 Anhydrous sodium sulphate

A-8.1.1.5 Sodium carbonate, anhydrous — see IS : 296-1974*.

A-8.1.1.6 Phosphoric acid — 1 : 2.

A-8.1.1.7 Sodium tungstate — 0.5 N. Dissolve 16.5 g sodium tungstate in 100 ml of water.

A-8.1.2 Procedure — Take 4 litres of the sample solution (see A-1.3) Adjust the pH to 4.5-5.5 by the addition of 50 percent sodium hydroxide solution or concentrated sulphuric acid. Add 80 ml sodium acetate solution and 16 ml 8-hydroxyquinoline reagent. Mix thoroughly, check the pH and maintain it again to 4.5-5.5.

Take one litre of the above solution in a separating funnel and extract the 8-hydroxyquinoline complex using 20 ml portions of chloroform. Combine the extracts and remove traces of water by adding anhydrous sodium sulphate. Transfer the chloroform extract to a platinum dish and evaporate chloroform at very low heat keeping the dish inside a well-ventilated fume chamber. The solution has a tendency to creep up the sides of the dish and hence, if necessary, carefully heat the sides of the dish with a burner.

When dry, place the dish in a muffle furnace at about 800 to 900°C and burn off all the organic matter. Add 5 g anhydrous sodium carbonate and fuse carefully at a temperature of 900 to 1 000°C, occasionally swirling the dish to cover the sides also. After about 10 to 15 minutes, cool the dish,

*Specification for sodium carbonate, anhydrous (second revision).

fill up two-third with water and warm gently to dissolve the melt. Transfer to a small beaker and filter through a Whatman filter paper No. 42. Neutralize the filtrate with concentrated sulphuric acid to pH 7. For blank, take 5 g of sodium carbonate in a beaker, dissolve in a small quantity of water and neutralize with concentrated sulphuric acid to pH 7. Add to both beakers enough sulphuric acid to make approximately 0.5 N solution and further add 5 ml phosphoric acid and 2.5 ml sodium tungstate solution. Keep the beakers on a hot-plate and boil gently for 2 minutes. Cool and make up to 50 ml with water. A pale yellow colour of the phosphotungstate would develop in the sample. Find out the percentage transmittance of the solution, taking the blank for 100 percent transmittance, in the photoelectric colorimeter using a cell of 5-cm light path and a blue 400 nm filter. Find out the corresponding optical density and from the standard graph (see A-8.1.3), calculate the equivalent vanadium content.

A-8.1.3 Preparation of Standard Graph — Ignite pure vanadium pentoxide at 500°C for an hour and cool it in a desiccator. Dissolve 1.785 0 g of this in a slight excess of sodium hydroxide solution. Neutralize with sulphuric acid and add a few drops in excess and finally dilute to 1 000 ml. One millilitre of this solution is equivalent to 1.0 mg of vanadium. Further, dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 10 micrograms of vanadium.

Take varying volumes of the diluted solution in 100-ml beakers so as to obtain 5, 10, 25, 50, 75 and 100 micrograms of vanadium. Dilute to about 40 ml and make acidic with concentrated sulphuric acid to make approximately 0.5 N. Add 5 ml phosphoric acid and 2.5 ml sodium tungstate solution to each. Boil for 2 minutes on a hot-plate, cool and dilute to 80 ml. Also take a blank using 40 ml water. Find out the percentage transmittance on a photoelectric colorimeter using blue 400 nm filter and calculate the corresponding optical densities. Plot the vanadium concentration against the corresponding optical density.

A-8.1.4 Calculation

$$\text{Vanadium content, parts per million,} = \frac{a}{4\,000}$$

in the sample solution

$$\text{Therefore, vanadium content } (X_1) = 3.125 \times \frac{a}{4\,000}$$

parts per million, in the salt

where

a = mass in micrograms of vanadium contained in 4 litres of the sample solution.

A-8.2 Estimation of Chromium

A-8.2.0 Principle— Chromium hexavalent is reduced to chromium trivalent by boiling with potassium iodide and sodium sulphite. The precipitate is then co-precipitated with aluminium hydroxide. The aluminium hydroxide is dissolved in 1 : 1 sulphuric acid, and chromium is oxidized to hexavalent forth by bromine water. Chromium is determined colorimetrically by reacting with diphenyl carbazide reagent, which gives a purple colour with chromium.

A-8.2.1 Reagents

A-8.2.1.1 Sulphuric acid— 1 : 1 (v/v) and 1 : 9 (v/v).

A-8.2.1.2 Phosphoric acid— 1 : 5 (v/v).

A-8.2.1.3 Aluminium solution— Dissolve 1 g of aluminium metal in hydrochloric acid and dilute to 1 litre.

A-8.2.1.4 Diphenyl carbazide reagent— Dissolve 0.25 g of diphenyl carbazide and 0.4 g of phthalic anhydride in 100 ml ethanol. The solution is stable for one month.

A-8.2.1.5 Bromine water.

A-8.2.1.6 Potassium iodide— 16 percent solution.

A-8.2.1.7 Sodium hydroxide solution— 30 percent.

A-8.2.1.8 Neutral sodium sulphite.

A-8.2.1.9 Potassium dichromate— See IS : 250-1964*.

A-8.2.2 Procedure— Take 2 litres of the sample solution (see A-1.3) in a 4-litre beaker, add 40 ml of potassium iodide solution, 40 ml sulphuric acid and 200 ml water. Boil for about 10 minutes. Add sodium sulphite, in small portions, to decolorize the solution. In case, the solution turns brown on further boiling, continue adding sodium sulphite till the decolorization is stable and brown colour of iodine does not reappear on boiling. Add 30 ml of aluminium solution and stir well. Further, add sodium hydroxide solution dropwise, stir the solution well and adjust the pH to 6.0-6.5. Allow to settle to complete precipitation. Siphon out the clear solution and collect the aluminium precipitate in a small beaker. Filter through a sintered glass crucible and wash once with hot water. Dissolve the precipitate by pouring 60 ml boiling sulphuric acid (1 : 9) into the crucible, apply suction after 2 to 3 minutes and again wash with hot water.

*Specification for potassium bichromate, technical and analytical reagent (revised).

Collect the filtrate separately in a 250 ml beaker. Add 4 ml bromine water and stir well. Decolorize the yellow colour with a few drops of sodium hydroxide solution and add 1 ml in excess. Boil for 5 minutes and add 1 : 1 sulphuric acid until the brownish colour appears. Add 2 ml of the acid in excess and boil again till the brownish colour disappears. Cool and add 1 ml phosphoric acid. Make up to 100 ml and divide into two parts. Keep one as a blank and in the other add 1 ml diphenyl carbazide solution. Make up both solutions to 100 ml each. Wait for 10 to 15 minutes for the full colour to develop and then find out the percentage transmittance on a photoelectric colorimeter using 460 nm green filter and calculate the corresponding optical density. From the standard graph (see A-8.2.3) find out the corresponding concentration of chromium.

A-8.2.3 Preparation of Standard Graph — Weigh 0.283 g of dried potassium dichromate, dissolve in a little water and dilute to one litre. One millilitre of this solution is equivalent to 0.1 mg chromium (as Cr). Further, dilute 10 ml of the above solution to 1 litre. One millilitre of the diluted solution is equivalent to 1 microgram of chromium. Take various volumes of the standard solution so as to give 5, 10, 15, 20 and 25 micrograms of chromium. Add 15 ml aluminium solution to each dilute to about 50 ml and add 30 ml 1 : 1 sulphuric acid and 2 ml bromine water. Further, add 30 percent sodium hydroxide dropwise till the colour disappears and add 2 drops in excess. Boil for 5 minutes and cautiously add 1 : 1 sulphuric acid till the bromine colour reappears and add 0.5 ml acid in excess. Again boil for about 20 minutes until bromine colour disappears. Check for bromine, cool and add 1 ml diphenyl carbazide solution and make up the volume to 100 ml. Let it stand for 15 minutes and then take the reading on a photoelectric colorimeter using green 460 nm filter and a glass cell with 5 cm light path. Find out the corresponding optical density. For adjusting 100 percent transmittance use 50 ml water and proceed in the same manner as above. Plot the optical densities against the corresponding chromium concentrations.

A-8.2.4 Calculation

$$\text{Chromium, parts per million, in} = \frac{a}{1000}$$

the sample solution

$$\text{therefore, chromium } (X_2), \text{ parts} = 3.125 \times \frac{a}{1000}$$

per million, in the salt

where

a = mass in micrograms of chromium contained in 1 litre of the sample.

A-8.3 Estimation of Molybdenum

A-8.3.0 Principle — Molybdenum is extracted as the 8-hydroxyquinoline complex and then converted into the sulphate, which is further reacted with alkali thiocyanate in the presence of a strong reducing agent like stannous chloride, to give a cherry red colour. The colour is extracted with an organic solvent and the intensity is measured in a photoelectric colorimeter.

A-8.3.1 Reagents

A-8.3.1.1 Anhydrous potassium pyrosulphate.

A-8.3.1.2 Dilute sulphuric acid — 10 percent (v/v).

A-8.3.1.3 Ferrous ammonium sulphate — 1 percent.

A-8.3.1.4 Potassium thiocyanate — 5 percent.

A-8.3.1.5 Stannous chloride — 10 percent. Dissolve 10 g stannous chloride in 20 ml 1:1 hydrochloric acid and dilute to 100 ml. Filter, if not clear.

A-8.3.1.6 Mixture of isoamyl alcohol and carbon tetrachloride — 1:1 .

A-8.3.1.7 Sodium molybdate — AR grade.

A-8.3.2 Procedure — Extract 2 litres of the sample solution (see A-1.3) with 8-hydroxyquinoline by the method described in A-8.1.2. Evaporate chloroform completely in a well-ventilated fume chamber and ignite at low temperature. Add 5 g of potassium pyrosulphate and fuse in a muffle furnace at 850°C, swirling the dish at intervals to spread the melt along the sides of the dish. Add sufficient quantity of dilute sulphuric acid to dissolve the melt and transfer to a separating funnel which has been dried in an oven. Add 1 ml ferrous ammonium sulphate solution, 10 ml of 5 percent potassium thiocyanate solution and mix thoroughly. Add 10 ml of 10 percent stannous chloride solution and make the solution to about 50 ml.

Shake this solution with 20 ml portions of isoamyl alcohol and carbon tetrachloride mixture, collect the alcoholic layer into a 50 ml standard volumetric flask and make up the volume to 50 ml. The extract should be clear. If turbid keep the flask in hot water for a few seconds. Carry out the blank using 1 : 1 mixture of isoamyl alcohol and carbon tetrachloride and adjust 100 percent transmittance on the photoelectric colorimeter, using a 5-cm light path cell and 460 nm filter. From the standard graph (see A-8.3.3) find out the molybdenum concentration.

A-8.3.3 Preparation of Standard Graph— Dissolve 0.282 g sodium molybdate in 1 000 ml water. Further, dilute 10 ml of the above solution to 1 000 ml. One millilitre of this solution is equivalent to 1 microgram of molybdenum. Take aliquot parts of the standard molybdenum solution so as to obtain 5, 10, 15, 20 and 25 micrograms of molybdeum. Add 5 ml of dilute sulphuric acid and dilute to about 40 ml. Add 1 ml ferrous ammonium sulphate, 10 ml potassium thiocyanate solution and 10 ml stannous chloride solution. Transfer to a separating funnel and extract with two 20 ml portions of 1 : 1 mixture of isoamyl alcohol and carbon tetrachloride. Combine extracts and make up to 50 ml with the same mixture. Find out the percentage transmittance using a photoelectric colorimeter with green 460 nm filter and a glass cell of 5-cm light path. Find out the corresponding optical density. Plot the optical densities against the molybdenum concentration on a graph paper.

A-8.3.4 Calculation

$$\text{Molybdenum content in the sample solution,} = \frac{a}{2\,000}$$

parts per million

$$\text{Therefore, molybdeum content (} X_3 \text{) in} = 3.125 \times \frac{a}{2\,000}$$

the salt, parts per million

where

a = mass in micrograms of molybdenum contained in 2 litres of the sample.

A-8.4 Calculate vanadium + Chromium + molybdenum as below:

$$(\text{Vanadium + Chromium + Molybdenum}), = X_1 + X_2 + X_3$$

parts per million in the salt

A-9. TEST FOR ALUMINIUM

A-9.0 Principle— Aluminium forms red coloured lake with aluminor reagent. It is then colorimetrically estimated.

A-9.1 Reagents

A-9.1.1 Buffer Solution— Dissolve 154 g of ammonium acetate and 6 ml of acetic acid in water and dilute to 1 litre.

A-9.1.2 Aluminon Reagent— 0.10 percent aqueous solution. If the solution is reddish, add a drop of ammonia and make it golden yellow.

A-9.1.3 Thioglycollic Acid— 10 percent.

A-9.1.4 Potassium Aluminium Sulphate.**A-9.1.5 Potassium Chloride Solution**—saturated.

A-9.2 Procedure—Take 50 ml of the sample solution (*see A-1.3*), acidity with hydrochloric acid, boil and filter. Neutralize the filtrate with ammonia to pH 6.8-7.2. Add 20 ml buffer solution, 1 ml thioglycollic acid solution and 1 ml aluminon reagent. Heat gently on a water-bath at 100°C for exactly 10 minutes, cool and make up to 100 ml.

For blank, take 50 ml water and follow the above procedure. Adjust 100 percent transmittance with blank using a photoelectric colorimeter with green 525 nm filter and a 5-cm light path glass cell. Find out the percentage transmittance of the sample and the corresponding optical density and from the standard graph (*see A-9.3*) find out the corresponding aluminium content in micrograms.

A-9.3 Preparation of Standard Graph—Dissolve 1.75 g potassium aluminium sulphate in water and make up to 1 litre. Dilute 10 ml of the above solution to one litre. One millilitre of this solution contains 1 microgram of aluminium (as Al). Take 50 ml saturated potassium chloride solution in six 250-ml beakers. Use one as a blank. Into the other beakers pipette out aliquots of standard aluminium solution so as to give 5, 10, 15, 20 and 30 micrograms of aluminium, and make up the volume to 70 ml with water. Add 20 ml buffer, 1 ml thioglycollic acid solution and 1 ml aluminon reagent to each beaker. Keep on a waterbath at 100°C for exactly 10 minutes, cool and make up to 100 ml with water.

Adjust the colorimeter to 100 percent transmittance with the blank on a photoelectric colorimeter using green 525 nm filter and a 5-cm light path cell and find out the percentage transmittance of the solution. Plot the corresponding optical densities against the aluminium concentration.

A-9.4 Calculation

$$\text{Aluminium content, parts per million,} = \frac{a}{50}$$

in the sample solution

$$\text{Therefore, aluminium content in the salt sample, parts per million} = 3.125 \times \frac{a}{50}$$

where

a = mass in micrograms of aluminium in 50 ml of the sample solution.

A-10. TEST FOR TITANIUM, COPPER AND NICKEL

A-10.0 Each metal is determined separately and the results are added up.

A-10.1 Determination of Titanium

A-10.1.0 Principle — Co-precipitation of titanium along with aluminium and the colorimetric determination of titanium with tiron (disodium 1, 2, dihydroxy benzene-3, 5 sulphonate)..

A-10.1.1 Reagents

A-10.1.1.1 Double distilled water

A-10.1.1.2 Aluminium solution

A-10.1.1.3 Concentrated hydrochloric acid See IS : 265-1976*.

A-10.1.1.4 Ammonia solution — 20 percent (m/m).

A-10.1.1.5 Concentrated sulphuric acid — See IS : 266-1977†.

A-10.1.1.6 Tiron — 10 percent solution.

A-10.1.1.7 Thioglycollic acid — 10 percent solution.

A-10.1.1.8 Buffer solution — Dilute 1.6 g of sodium acetate and 60 g acetic acid to 1 litre with double distilled water.

A-10.1.1.9 Potassium titanuim oxalate.

A-10.1.2 Procedure — Take 2 litres of the sample solution (see A-1.3) and proceed in the same manner as for chromium (see A-8.2.2), till the aluminium precipitate is dissolved in 50 ml boiling concentrated sulphuric acid. Cool and make up to 100 ml. Make two parts of 50 ml each.

In both, add 10 ml thioglycollic acid solution. Keep one beaker for the blank. In the other beaker add 5 ml tiron solution. Adjust the pH of the solution in both the beakers to 3-4 by adding ammonia solution. Finally, add 10 ml buffer solution and dilute to 100 ml with double distilled water. Use the blank to adjust 100 percent transmittance and take the percentage transmittance of the sample solution on a photoelectric colorimeter, using blue 410 nm filter and a cell with 5-cm light path. Find out the corresponding optical density and read out the titanium concentration corresponding to the optical density from the standard graph (see A-10.1.3)

*Specification for hydrochloric acid (second revision)

†Specification for sulphuric acid (second revision)

A-10.1.3 Preparation of Standard Graph - Weigh 3.687 5 g potassium titanium oxalate into a beaker. Add 15 g ammonium sulphate and 200 ml of concentrated sulphuric acid. Boil for 5 to 10 minutes, cool and make up to one litre. Further, dilute 10 ml of this solution to 1 000 ml. One millilitre of this solution contains 5 micrograms of titanium. Pipette out aliquots of the standard titanium solution containing 5, 10, 15, 20 and 25 micrograms titanium in 250-ml beakers. To each beaker add 15 ml 1 : 1 sulphuric acid, 15 ml aluminium solution and add 25 ml water. Then add 10 ml thioglycolic acid and 5 ml tiron solution to each beaker. Adjust the pH of the solution to 3-4 with ammonia or sulphuric acid. Finally add 10 ml buffer solution and make up to 100 ml. Find out the percentage transmittance using 410 nm blue filter and a glass cell of 5-cm light path. For adjusting 100 percent transmittance, use 50 ml water, treated in the same manner as above. Find out the corresponding optical densities and plot the titanium concentration against the optical densities.

A-10.1.4 Calculation

$$\text{Titanium content, parts per million,} = \frac{a}{1\,000}$$

in the sample solution

$$\text{Therefore, titanium content (} X_1 \text{) in the salt, parts per million} = 3.125 \times \frac{a}{1\,000}$$

where

a = mass in micrograms of titanium in one litre of sample solution.

A-10.2 Determination of Copper

A-10.2.0 Principle — Copper forms a complex with sodium diethyl dithiocarbamate solution, which is soluble in chloroform giving a yellow coloured solution which is determined colorimetrically.

A-10.2.1 Reagents

A-10.2.1.1 Ammonium citrate solution Take 210 ml concentrated ammonia solution and 150 ml water in a 500-ml beaker. Add, while stirring, 200 g of citric acid, cool and make the contents slightly alkaline with ammonia. Add 5 ml sodium diethyldithiocarbamate solution and extract with 10 ml portions of chloroform till the chloroform layer becomes water white. Dilute to 500 ml with water.

A-10.2.1.2 Sodium diethyldithiocarbamate solution — Take 0.1 g of sodium diethyldithiocarbamate in 100 ml volumetric flask, dissolve in water and dilute to 100 ml. Filter, if not clear, and store in a brown bottle. The solution is stable for about a week. It shall be kept at a cool place.

A-10.2.1.3 Ethylene diamine tetra acetate, sodium salt—Dissolve 20 g of ethylene diamine tetra acetate, sodium salt in 500 ml of water.

A-10.2.1.4 Phenolphthalein indicator.

A-10.2.1.5 Chloroform—See IS : 5296-1969*.

A-10.2.1.6 Ammonia solution—10 percent.

A-10.2.1.7 Copper sulphate (CuSO₄.5H₂O).

A-10.2.2 Procedure—Take 100 ml of the sample solution (see A-1.3) in a dry separating funnel. Add 5 ml ammonium citrate, 20 ml EDTA solution and 2 drops of phenolphthalein indicator solution. Shake vigorously and add ammonia solution till the solution in the funnel turns very slightly pink. Further, add 5 ml of sodium diethyldithiocarbamate solution and mix well. Extract the complex with 10 ml portion of chloroform till the chloroform layer becomes water white and make up to 50 ml. Use pure chloroform for 100 percent transmittance adjustment and find out the percentage transmittance of the sample, using a photoelectric colorimeter, with blue, 560 to 600 nm filter and a 5-cm light path glass cell. Find out the corresponding optical density and from the standard graph (see A-10.2.3.), read the copper concentration.

A-10.2.3 Preparation of Standard Graph—Dissolve 0.392 8 g of copper sulphate (CuSO₄.5H₂O) in a little water. Add 8 ml concentrated hydrochloric acid and dilute to one litre. One millilitre of this solution contains 0.1 milligram of copper. Further, dilute 10 ml of the above solution to 1 000 ml with water. One millilitre of the diluted solution contains 1 microgram of copper. Pipette out aliquots of standard copper solution so as to contain 5, 10, 15, 20 and 25 micrograms of copper. Dilute to about 100 ml with water and follow the procedure described for the sample (see A-10.2.2). Plot the optical densities against the copper concentration on a graph paper.

A-10.2.4 Calculation

$$\text{Copper content, parts per million,} = \frac{a}{100}$$

$$\text{Therefore, copper content (} X_2 \text{) in the salt, parts per million} = 3.125 \times \frac{a}{100}$$

where

a = mass in micrograms of copper in 100 ml of the sample.

*Specification for chloroform, technical and analytical.

A-10.3 Estimation of Nickel

A-10.3.0 Principle — Nickel forms a red coloured complex with dimethyl glyoxime which is soluble in chloroform. It can then be determined colorimetrically.

A-10.3.1 Reagents

A-10.3.1.1 Sodium citrate solution — 10 percent.

A-10.3.1.2 Dilute ammonia solution — 1 : 20 (*v/v*)

A-10.3.1.3 Dimethyl glyoxime reagent — 1 percent solution in ethanol.

A-10.3.1.4 Chloroform.

A-10.3.1.5 Hydrochloric acid — 0.5 N.

A-10.3.1.6 Bromine water.

A-10.3.1.7 Ammonium nickel sulphate [(NH₄)₂NiSO₄.6H₂O].

A-10.3.2 Procedure — Take 50 ml of the sample solution (see A-1.3) in a separating funnel, add 5 ml of sodium citrate solution and make the contents slightly alkaline with ammonia solution. Add 2 ml dimethyl glyoxime solution and extract the complex formed with chloroform using 5 ml quantities three to four times.

Collect the chloroform layers in a separating funnel. Add 5 ml hydrochloric acid and shake vigorously. Draw off the chloroform layer and collect the acid layer in a 50-ml volumetric flask. Add 10 drops of bromine water and let stand for a few minutes. Add liquor ammonia till the bromine colour disappears and add 1 ml in excess. Cool to below 30°C, add 1 ml dimethyl glyoxime reagent and dilute to 100 ml with water.

For blank, take 50 ml water in a separating funnel, to this add 5 ml hydrochloric acid, 10 drops of bromine water and let it stand for a few minutes. Add concentrated ammonia solution till the bromine colour disappears. Cool to below 30°C, add 1 ml dimethyl glyoxime reagent and make up the volume to 100 ml with water. Adjust 100 percent transmittance on the photoelectric colorimeter with this solution using a 465 nm filter and a 5 cm light path glass cell. Find out the percentage transmittance of the sample solution and calculate the corresponding optical density. From the standard graph (see A-10.3.3) read out the concentration of nickel.

A-10.3.3 Preparation of Standard Graph — Dissolve 0.508 g of ammonium nickel sulphate [$(\text{NH}_4)_2 \text{NiSO}_4 \cdot 6\text{H}_2\text{O}$] in a little water and dilute to one litre. Dilute 10 ml of this solution to 100 ml. One millilitre of this solution contains one microgram of nickel. Pipette out aliquots of the standard nickel solution so as to give 5, 10, 20, 30 and 50 micrograms of nickel. Dilute to 50 ml with water and follow the procedure as given for the sample (see A-10.3.2). Plot the optical densities obtained against the concentration of nickel on a graph paper.

A-10.3.4 Calculation

$$\text{Nickel content, parts per million,} = \frac{a}{50}$$

$$\text{Therefore, nickel content } (X_3), \text{ parts per million, in the salt} = 3.125 \times \frac{a}{50}$$

where

a = mass in micrograms of nickel contained in 50 ml of sample.

A-10.4 Calculate titanium + copper + nickel as below;
(Titanium + Copper + Nickel),
parts per million, in the salt $= X_1 + X_2 + X_3$

A-11. DETERMINATION OF GRIT

A-11.1 Procedure — Use the water insoluble residue obtained in A-4.1. Brush the residue into a beaker, treat it with a few millilitres of aqua regia and warm gently. Cool and dilute the solution, and filter through a filter paper. Wash the residue on the filter paper free of chloride, dry and ignite the filter paper in a silica crucible; cool and weigh. Brush the ignited residue on a 6.3 micron IS Sieve and weigh the material retained on the sieve.

A-12. DETERMINATION OF ALKALINITY

A-12.1 Reagents

A-12.1.1 Standard Hydrochloric Acid — 0.1 N.

A-12.1.2 Methyl Orange Indicator — Dissolve 0.5 g of methyl orange in 100 ml of water.

A-12.2 Procedure — Dissolve about 25 g of dried sample preserved in A-1.1 in about 400 ml of water, filter and wash the residue free from soluble salts. Collect the filtrate and washings. Take 100 ml from the filtrate and washings. Add two or three drops of methyl orange indicator. Titrate the solution with standard hydrochloric acid to colour change from orange to pinks. Note down the burette reading.

A-12.3 Calculation

$$\text{Alkalinity (as Na}_2\text{CO}_3\text{), percent} = \frac{V \times N \times 5.3}{M}$$

where

V = volume in ml of standard hydrochloric acid,

N = normality of standard hydrochloric acid, and

M = mass in g of prepared sample taken for the test.

APPENDIX B

(Clause 5.1)

SAMPLING OF COMMON SALT FOR CHEMICAL INDUSTRIES**B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.0 In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place, exposed to weather.

B-1.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 For drawing a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass containers.

B-1.5 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and year of manufacture.

B-2. SCALE OF SAMPLING**B-2.1 Lot**

B-2.1.1 *For Heaps and Wagons* — Each individual heap or wagon shall constitute a separate lot.

B-2.1.2 *For Packages*

B-2.1.2.1 All the packages in a single consignment of common salt of the same grade, drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute a separate lot.

B-2.1.2.2 The number of packages (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF PACKAGES TO BE SELECTED FOR SAMPLING

LOT SIZE (1) N	No. OF PACKAGES TO BE SELECTED (2) n
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.1.2.3 The packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed.

B-2.1.2.4 Samples shall be tested for each lot, for ascertaining conformity of the material to the requirements of this specification.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 For Wagons and Lorries — From each wagon or lorry, draw with an appropriate sampling instrument, several portions of the material from different parts, for example, front, middle and back, and at different depths. Collect about 25 kg of the sample in this way.

B-3.2 For Heaps — In case of heaps, draw samples from different heights, depths and intervals. The quantity of sample drawn from each height should be proportional to the quantity of salt present in that section. Collect about 25 kg of the sample in this way.

B-3.3 Preparation of Composite Sample — Spread out the samples collected and mixed as above (see B-3.1 and B-3.2) on a level, clean and hard surface. Flatten it out first and then scoop the mass together into a cone. Flatten the heap again and divide into four portions. Remove the two diagonally opposite quadrants. Mix the remaining two portions well again, form into a cone and repeat this process until finally about 2 kg of the material is left. This shall be called the composite sample. This composite

*Methods for random sampling.

sample shall be divided into three parts, one for the purchaser, another for the supplier and the third shall be used as a referee sample. Transfer the samples to separate, clean and dry sample containers and label with full identification particulars.

B-3.4 Packages

B-3.4.1 From each of the packages selected according to **B-2.1.2.3**, withdraw portions of about 500 g each from different parts and collect about 1 kg of sample from each package.

B-3.4.2 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 500 g. The composite sample shall be divided into three parts, one for the purchaser another for the supplier and the third shall be used as a referee sample.

B-3.5 The referee test samples consisting of composite samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, and shall be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of all the characteristics given in Table 1 shall be performed on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 The lot shall be considered as conforming to the specification if the test results for the tests carried on the composite test sample satisfy the corresponding requirements given in 3 and Table 1.

IS : 797 - 1982

(Continued from page 2)

<i>Members</i>	<i>Representing</i>
SHRI K. L. SAHNI	Salt Commissioner, Jaipur
SHRI D. V. KIRTIKAK (<i>Alternate</i>)	
SHRI G. P. THACKER	The Indian Salt Manufacturers Association, Bombay
SHRI DHIRAJLAL G. SHAH (<i>Alternate</i>)	
SHRI T. P. VENKATRAMAN	Standard Alkali, Chemicals Division, Bombay
SHRI B. R. WALAVALKAR	Saurashtra Chemicals, Porbandar
SHRI L. K. ??? (<i>Alternate</i>)	

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhawan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones. 23230131, 23233375, 23239402 Fax 91+011 23239399, 23239382
E - Mail: bis@vsnl.com website <http://www.bis.org.in>

Central Laboratory:

Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010 27700 32

Regional Offices:

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001	560 13 48
Peenya Industrial Area, 1 st Stage, Bangalore-Tumkur Road, BANGALORE	839 49 55
Commercial-cum-Office Complex, Opp. Dushera Maidan, E-5 Arera Colony,	242 34 52
Bittan Market, BHOPAL 462016	
62-63, Ganga Nagar, Unit VI, BHUBANESHWAR 751001	240 31 39
5 th Floor, Kovai Towers, 44 Bala Sundaram Road, COIMBATORE 641018	221 01 41
SCO 21 Sector 12, Faridabad 121007	229 2175
Savitri Complex, 116 G.T Road, GHAZIABAD 201001	286 1498
Plot No A-20-21, Institutional Area, Sector 62, Goutam Budh Nagar, NOIDA-201307	240 22 06
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, Apurba Sinha Path,	254 11 37
GUWAHATI 781003	
5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	2320 10 84
E-52, Chittaranjan Marg, C-Scheme, JAIPUR 302001	237 38 79
117/418 B, Sarvodaya Nagar, KANPUR 208005	221 82 92
Sethi Bhawan, 2 nd Floor, Behind Leela Cinema, Naval Kishore Road,	221 56 98
LUCKNOW 226001	
NIT Building, Second Floor, Gokulpur Market, NAGPUR 440010	252 51 71
Mahabir Bhavan, 1 st Floor, Ropar Road, NALAGARH 174101	22 14 51
Patliputra Industrial Estate, PATNA 800013	226 28 08
First Floor, Plot Nos 657-660, Market Yard, Gultkdi, PUNE 411037	426 86 59
"Sahajanand House" 3 rd Floor, Bhaktinagar Circle, 80 Feet Road,	237 82 51
RAJKOT 360002	
TC. No. 14/1421, University P.O. Palayam, THIRUVANANTHAPURAM 695034	232 21 04
1 st Floor, Udyog Bhavan, VUDA, Siripuram Junction, VISHAKHAPATNAM-03	271 28 33
Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, KOLKATA 700072	22 12 6215
Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	2309 65 28